FY95 End of Fiscal Year Letter
(01 Oct 1995 - 30 Sep 1996)

ONR CONTRACT INFORMATION

Contract Title: Ceramic Fibers and Ceramic-Matrix Composites for Ultrahigh Temperature Applications

Performing Organization: University of Florida

Principal Investigator: M.D. Sacks

Contract Number: N00014-93-1-0853

R & T Project Number: 4313410---04

ONR Scientific Officer: Dr. Steven G. Fishman

Enclosure (1)

A. Scientific Research Goals

The overall objective of this ASSERT project is to establish processing-microstructure-property relations for SiC fibers prepared using novel pre-ceramic polymer blends and selected processing additives. Under the ASSERT parent award, it was demonstrated that SiC fibers with improved thermomechanical stability (e.g., compared to commercial fibers such as Nicalon® and Hi-Nicalon®) can be fabricated using blends of organosilicon (pre-ceramic) polymers. Such fibers are of interest for application in high temperature composite materials. In the present ASSERT project, the objective is to relate the chemical/structural characteristics of selected pre-ceramic polymer blends with the SiC fiber microstructure and mechanical properties. Another objective is to determine the effect of selected processing additives (i.e., boron- and aluminum-containing sintering aids and polysilazane fiber processing aids) on microstructure and mechanical properties.

B. Significant Project Results

In previous studies, SiC fibers with near-stoichiometric composition, low oxygen content, and improved thermomechanical stability were prepared using boron (≤0.75%) as a sintering aid. In this program, the role of other additives on fiber processing and properties has been investigated. The fibers were prepared using essentially the same procedures as in previous work. Fibers were fabricated by dry spinning of concentrated solutions of a high-molecular-weight polycarbosilane. An aluminum-containing additive was investigated as an alternative sintering aid. The role of polyvinylsilazane as a fiber processing aid was also investigated.

All polymers were synthesized in the PI's laboratory. Aluminum was doped into the fiber as a polymer prepared from a organometallic precursor. Fibers prepared with a mixture of boron (~0.5-1.0%) and aluminum (~0.25%) dopants showed similar properties to those

reported previously for fibers prepared with boron alone. Scanning electron microscopy (SEM) observations showed that fibers had fine diameters (~10-15 μm), smooth outer surfaces, and minimal residual porosity. X-ray diffraction (XRD) showed that fibers were mostly β-SiC with a small amount of α-SiC. Electron microprobe analysis (EMA) indicated that fibers were slightly carbon-rich. Fibers had very low residual oxygen and nitrogen contents ($\leq$0.1 wt%). The average fiber density was >3.1 g/cm^3, i.e., >97% of the theoretical value of ~3.21 g/cm^3 for fully dense (pore-free), stoichiometric SiC. Fiber room temperature tensile strengths were typically on the order of 2.5 GPa.

Fibers were also prepared with aluminum doping alone (i.e., no boron additive). The properties of the fibers depended upon the amount of the aluminum. Fibers prepared with ~0.25% aluminum had poor tensile strength (<1.5 GPa). SEM observations indicated that the fiber strength was low due to the presence of porosity and larger grains in the fibers. Fibers prepared with higher aluminum contents (~0.5-1.0%) had improved tensile strength (~1.5-2 GPa). SEM observations indicated that fibers had less porosity and smaller grains. Fiber prepared with higher aluminum contents (~0.5-1.0%) and low boron contents (~0.25%) had improved tensile strength (~2-2.5 GPa).

The role of polyvinylsilazane (PSZ) as a processing aid was investigated. Solutions prepared with and without PSZ were characterized by rheological measurements, contact angle and surface tension measurements, and fiber spinning behavior. Fiber spinnability was greatly improved for solutions prepared with PSZ. This also resulted in significant improvements in fiber tensile strengths after heat treatment.

C. Plans for Next Year's Research

This project was concluded in June 1996.

D. List of Publications/Reports/Presentations

1. Papers Published in Refereed Journals and in Refereed Conference Proceedings

M.D. Sacks, A.A. Morrone, G.W. Scheiffele, and M. Saleem, "Characterization of Polymer-Derived Silicon Carbide Fibers with Low Oxygen Content, Near-Stoichiometric Composition, and Improved Thermomechanical Stability," Ceram. Eng. Sci. Proc., 16 [4] 25-35 (1995).

M.D. Sacks, G.W. Scheiffele, M. Saleem, G. Staab, T.J. Williams, and A.A. Morrone, "Polymer-Derived Silicon Carbide Fibers with Near-Stoichiometric Composition and Low Oxygen Content"; pp. 3-10 in Ceramic Matrix Composites - Advanced High Temperature Structural Materials, edited by R.A. Lowden, M.K. Ferber, J.R. Hellmann, S.G. DiPietro, and K.K. Chawla, Mat. Res. Soc. Symp. Proc., Vol. 365, Materials Research Society, Pittsburgh, PA, 1995.

2. Non-Refereed Publications and Published Technical Reports

None

3. Presentations

a. Invited

"Silicon Carbide Fibers", German Aerospace Research Establishment (DLR), Cologne, GERMANY, June 18, 1996

b. Contributed

None

4. Books (and sections thereof)

None

E. LIST OF HONORS/AWARDS

Name of Person Receiving Award	Recipient's Institution	Name, Sponsor and Purpose of Award
M.D. Sacks	Univ. of Florida	Richard M. Fulrath Pacific Memorial Award (awarded by Japan/U.S. Fulrath Committee for scientific achievement by ceramist under the age of 45)

F. Participants in the ASSERT Project

Thomas J. Williams - graduate student

Gregory J. Klempel - undergraduate student

G. Other Sponsored Research During FY95

1. Title: Seeded Grain Growth for Textured Silicon Carbide and Mullite Fibers

   Sponsor: U.S. Air Force (AFOSR)

   Project Starting/Ending Dates: 09/01/94 - 08/31/97

   Funding for 09/01/95-08/31/96: $128,854

   P.I. Time Charged: 17%

2. Title: High Performance Silicon Carbide Fibers for High Temperature Composites

   Sponsor: BIRL (Northwestern University)/IHPTET Consortium

   Project Starting/Ending Dates: 08/95 - 04/96

   Funding for 08/95-04/96: $68,000

   P.I. Time Charged: 0%

3. Title: Silicon Carbide Multifilament Program

   Sponsor: 3M Co.

   Project Starting/Ending Dates: 03/96 - 03/97

   Funding for 03/96-03/97: $325,000

   P.I. Time Charged: 25%

H. SUMMARY OF FY95 PUBLICATIONS/PATENTS/PRESENTATIONS/HONORS/PARTICIPANTS (Number Only)

		ONR*	non ONR
a.	Number of Papers Submitted to Refereed Journal but not yet published:	2	1
b.	Number of Papers Published in Refereed Journals:	4	1
c.	Number of Books or Chapters Submitted but not yet Published:	0	0
d.	Number of Books or Chapters Published:	0	0
e.	Number of Printed Technical Reports and Non-Refereed Papers:	1	0
f.	Number of Patents Filed:	1	0
g.	Number of Patents Granted:	0	0
h.	Number of Invited Presentations at Workshops or Professional Society Meetings:	4	2
i.	Number of Contributed Presentations at Workshops or Professional Society Meetings:	3	3
j.	Honors/Awards/Prizes for Contract/Grant Employees: (selected list attached)	0	1
k.	Number of Graduate Students and Post-Docs Supported at least 25% this year on contract grant:	1	5
	Grad Students: TOTAL	1	5
	Female	0	0
	Minority	0	0
	Post Doc: TOTAL	0	0
	Female	0	0
	Minority	0	0
l.	Number of Female or Minority PIs or CO-PIs		
	New Female	0	0
	Continuing Female	0	0
	New Minority	0	0
	Continuing Minority	0	0

* **Includes ASSERT parent award**